Patent Application of

Jiang Lu and Samuel F. Azlein

For

TITLE: AEROSOL SPLITTER FOR ELSD

CROSS-REFERENCE TO RELATED APPLICATIONS Provisional Patent Application Number 60/457,637 filed on 03/26/2003

FEDERALLY SPONSORED RESEARCH Not Applicable

SEQUENCE LISTING OR PROGRAM Not Applicable

BACKGROUND OF THE INVENTION—FIELD OF INVENTION

This invention relates to division of an aerosol cloud formed by a nebulizer within an Evaporative Light Scattering Detector.

BACKGROUND OF THE INVENTION

An Evaporative Light Scattering Detector (ELSD) is an analytical instrument for detecting and quantifying samples that have been separated by any of a variety of chromatographic methods. Such methods include but are not limited to High Performance Liquid Chromatography (HPLC), Supercritical Fluid Chromatography (SFC), and Gel Permeation Chromatography (GPC).

The simplest embodiment of an ELSD has a nebulizer, a heated zone or drift tube, a light source, and an amplifier, which converts scattered light into an electrical signal. In operation, the column effluent, which contains both the mobile phase and analyte, is first sent to the nebulizer. The nebulizer transforms the effluent into an aerosol cloud, and propels the cloud into the instrument. As the aerosol cloud enters the drift tube, which is heated, the more volatile mobile phase evaporates, leaving a cloud of analyte particles. These particles scatter light from the light source. The scattered light is amplified by a photo-multiplier tube, photo-diode, or similar device into a useable electrical signal.

This simplest embodiment, referred to as "full flow" in Patent 6,629,605 and illustrated as Figure 1 in same, has many limitations. Principally, it will only evaporate

modest amounts of volatile mobile phases. While limited, full flow instruments are quite sensitive within their permissible operating range. The ALLTECH MODEL 500 is an example of such an instrument. To address the problem of limited evaporative power, several solutions have been tried.

One solution, available on instruments form SEDERE involves a nebulization chamber (spray chamber) placed between the nebulizer and drift tube. The nebulized effluent is divided in this chamber by impaction/condensation on the walls of the chamber. The chamber is geometrically constructed such that larger aerosol droplets hit the wall and run out a drain, while smaller aerosol droplets follow gas flow through the spray chamber and enter the drift tube. Patent 6,229,605 refers to these instruments as "split-flow" designs. As pointed out in the above-cited patent, split-flow instruments accommodate high effluent flow rates and difficult to evaporate mobile phases, but they do not always pass on enough aerosol to maximize sensitivity.

A second solution is available from ALLTECH ASSOCIATES, as a MODEL 2000. This instrument has a splitter that can be turned on or off, as described in the above-cited patent. Turning the splitter on involves rotating a plate impactor perpendicular to aerosol flow. Large aerosol droplets hit the plate, condense, and exit a drain. Smaller aerosol droplets traverse the annular space between impactor and wall, and continue on to the drift tube. Turning the impactor plate parallel to the aerosol flow

essentially removes it from the instrument, which then becomes "full flow". Thus the instrument is easily converted from "split-flow" to "full flow" modes.

The above-described design has advantage over earlier art, but still has objectionable limitations. Namely, (1) it has no intermediate settings, and (2) it relies on mechanical means (motor, solenoid or the like) to move the impactor.

As a detector for chromatography, an ELSD may reasonably be expected to handle a wide variety of effluent flow rates and mobile phase compositions. An on/off design, as described in Patent 6,229,605, handles the extremes adequately, but cannot be optimized for moderate flow rates, or moderately difficult to evaporate mobile phases. Also, motors, solenoids, shaft seals and linkages are possible sources of mechanical failure.

BACKGROUND OF INVENTION--OBJECTS AND ADVANTAGES

As can be seen from the above discussion, the prior art of aerosol splitters does not meet the needs of an ELSD user.

Several objects and advantages of the present invention are:

(a) to provide an aerosol splitter that is smoothly variable over a wide dynamic range. This allows an ELSD to be optimized for the wide variety of conditions encountered in chromatography.

(b) to provide an aerosol splitter without complex mechanical components.

(c) to provide an aerosol splitter whose variable split ratio is under user control.

(d) to provide an aerosol splitter capable of smoothly changing during a gradient run in chromatography. Gradient separations use more than one solvent in time-programmed compositions. Each composition requires a unique setting for the best instrument sensitivity.

Still further objects and advantages will become apparent from a consideration of the ensuing description and drawings.

SUMMARY

The present invention utilizes a combination of geometry and thermal technique to split an aerosol cloud. A user, by changing only one temperature, can vary the split over a wide dynamic range.

DRAWINGS—FIGURES

Fig. 1 shows the splitter assembled, and in perspective view.

Fig. 2a and 2b show exploded views of the splitter, illustrating how parts interact.

Fig. 3a and 3b show the spray chamber in perspective and in cut-away.

Fig. 4a and 4b shows the nebulizer holder in perspective and in cut-away.

Fig. 5 shows the splitter assembled in a typical ELSD configuration.

DRAWINGS—Reference Numerals

12 Nebulizer

14 Nebulizer Holder

16 Spray Chamber

18 Clamshell, left

16a Straight Section

16b Curved Section

16c Drain

20 Clamshell, right

22 Thermoelectric Plate

23 Fan

22a Heat Sink

24 Drift Tube

26 Light Source

28 Light Trap

32 Exhaust Tube

DETAILED DESCRIPTION—PREFERRED EMBODIMENT

With principal reference to Fig. 2a, a preferred embodiment of the aerosol splitter is illustrated. The device comprises a nebulizer **12**. The nebulizer is preferably an Elemental Scientific, Inc. model PFA-LC-2. The nebulizer **12** inserts into a Nebulizer Holder **14**. The Nebulizer Holder **14** makes a gas tight connection between the Nebulizer **12** and the Spray Chamber **16.** Figures 4a and 4b show the Nebulizer Holder in greater detail. Figure 4b shows the grooves, which hold O-rings. These O-rings (not shown) make gas tight seals around both Nebulizer **12** and Spray Chamber **16**.

The Spray Chamber **16** ultimately attaches to a Drift Tube **24** within the completed instrument, as illustrated in Fig. 5. To facilitate the attachment, the Spray Chamber **16** has a groove in the flange, which accommodates an O-ring (not shown). The O-ring allows for a gas tight connection while providing a measure of thermal isolation. Thermal isolation allows the Spray Chamber **16** and Drift Tube **24** to operate at different temperatures.

The Spray Chamber **16** is preferably constructed from 316 stainless steel, providing both good resistance to corrosion and good heat transfer. The Spray Chamber **16** is firmly sandwiched between two Clamshells **18** and **20**. These Clamshells **18** and **20** have a cavity milled within that matches the configuration of the Spray Chamber **16**.

Firmly connected to the right Clamshell **20** is a Thermoelectric Plate (also known as a peltier device) **22**. The Thermoelectric Plate **22** is preferably a TE Technology, Inc. Model CP-2721. Depending on the polarity of Direct Current (DC) electricity supplied to the Thermoelectric Plate **22**, and in the presence of sufficient airflow across the Heat Sink **22a** of the Thermoelectric Plate **22**, said Thermoelectric Plate **22** will become either a heater or a cooler. An electric Fan **23** supplies sufficient airflow in this embodiment. In turn, the Thermoelectric Plate **22** will heat or cool the clamshells **18** and **20** and the Spray Chamber **16** located within them.

As illustrated in Fig. 3 and 3a, the Spray Chamber **16** has a Straight Section **16a**. The Straight Section **16a** is followed by a Curved Section **16b**. When oriented as shown in Fig. 3, the Curved Section **16b** has a Drain **16c** located at its lowest point.

When particles exit the nebulizer at a high velocity, they have a momentum based on that velocity and upon their mass. Given the same velocity, larger particles will have more momentum than smaller particles. Above a critical momentum value, a particle will impact the wall of the Curved Section **16b**. Below this value the particle will follow gas flow around the Curved Section **16b** and enter the Drift Tube **24**.

When the Thermoelectric Plate **22** is in cooling (sub-ambient) mode, the spray Chamber **16** is also cooled due to its intimate mechanical contact. A cool Spray Chamber **16** will tend to condense the aerosol droplets exiting the nebulizer. These new larger

droplets will tend to have their momentum carry them into Curved Section **16b**. They then condense and exit the Drain **16c**.

When the Thermoelectric Plate **22** is in heating mode, the Spray Chamber **16** is also heated due to its intimate mechanical contact. A heated Spray Chamber **16** will partially evaporate the aerosol droplets exiting the nebulizer. These new smaller droplets will tend to be carried around the Curved Section **16c** by the airflow, where they will enter the Drift Tube **24**.

After evaporation in the Drift Tube **24**, the analyte particles pass in front of light emitted from Light Source **26**. Most of the emitted light passes through and is trapped by Light Trap **28**, however the analyte will scatter some of the incident light. The scattered light is detected and amplified by Amplifier **30**. Evaporated mobile phase and analyte exit via Exhaust Tube **32**.

The most preferred embodiment of the present invention may have the following dimensions for the Spray Chamber **16**.

Outer Diameter 1" with a small section at the straight end reduced to .95"

Inner Diameter .85"

Curved Section 90 degree arc, with radius of 2.5" at center.

Straight Section 4.5"

OPERATION

From the above discussion it can be appreciated by those skilled in the art that a curved Spray Chamber **16** can act as a controllable aerosol splitter when its walls undergo controlled temperature excursions. The splitting can be varied over a wide range by changing only one temperature. This invention places smoothly variable split ratios under operator control, without the use of complex mechanical mechanisms.

To use the splitter, the ELSD into which it is built will have both a temperature display with a user definable set point, and appropriate Thermoelectric Plate **22** drive circuitry. When a set point is entered above the present temperature of the Spray Chamber **16**, the drive circuitry will power the Thermoelectric Plate **22** in such a way that it is a heater. Conversely, when the set point is below the present temperature of the Spray Chamber **16**, the drive circuitry will power the Thermoelectric Plate **22** in such a manner that it is a cooler.

The user may decide whether to send more or less aerosol to the Drift Tube **24** by monitoring the baseline of the instrument for a given Drift Tube **24** temperature. Temperature of the Spray Chamber **16** can be increased until it is no longer possible for the Drift Tube **24** to successfully evaporate the aerosol, resulting in a noisy baseline. Reducing the Spray Chamber **16** temperature slightly from this point gives greatest instrument sensitivity.

In operation, the illustrated Spray Chamber **16** can divert from 1% to 99% of many mobile phases to drain, when operated between 80°C, and 0°C. This has been shown to be sufficient for a wide range of effluents and flow rates.

Conclusions, Ramifications and Scope of Invention

The reader can see that the invention provides a way of optimizing the aerosol split ratio of an ELSD for maximum instrument response, and for a wide variety of mobile phase types and flow rates. Previous art has either not allowed the split ratio to be varied under user control, or has provided only extreme settings.

While the above description contains many specificities, these should not be construed as limitations on the scope of the invention, but rather as an example of one preferred embodiment. Without departing from the invention, many other variations are possible.

For example, the Nebulizer Holder **14** may have many different constructions. It could have threaded portions that compress O-rings, instead of static seals. The overall shape of the inner chamber could also be modified without departure from the invention.

The Nebulizer **12** could be of many different types. The illustrated type is a concentric flow pneumatic nebulizer, but cross flow, non-concentric, and ultrasonic could all be employed.

The Spray Chamber **16** could be of different shape or construction. No straight section is required if gas velocity from the Nebulizer **12** is suitable. The Curved Section **16b** can be of different arc length and radius. The Curved Section **16b** could be in coiled form.

The Thermoelectric Plate **22** could be replaced with other means of heating and cooling, such as circulating a temperature controlled liquid through embedded or external passages.

Other means of attaching the Spray Chamber **16** to the Drift Tube **24** are possible.

Since other analytical devices and processes also use nebulization and desolvation as core processes (i.e. mass spectroscopy), the invention could have application other than within an ELSD.

Therefore, the scope of the invention should be determined not by the illustrated embodiment, but by the appended claims and their legal equivalents.